EXPERIMENTAL INVESTIGATION OF NITRIDING ON THE MICROSTRUCTURAL, MECHANICAL AND CORROSION PROPERTIES OF ADDITIVELY MANUFACTURED 17-4 PH STAINLESS STEEL

A SYNOPSIS

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1. INTRODUCTION

Precipitation Hardened (PH) Stainless Steels are being used in many applications such as aerospace, marine, construction, chemical, petroleum, and oil industries, etc. These alloys possess excellent mechanical properties including strength, hardness, wear, and moderate corrosion resistance. Generally, PH steels showed poor ductility and toughness. In order to improve the strength and toughness, elements such as nitrogen, copper, niobium and chromium are added and that forms precipitates on aging treatment. Chromium content presents in PH steel form a protective Cr_2O_3 oxide layer which protects the material from corrosion.

Manufacturing of complex geometrical metal parts in the field of aviation, automotive and constructive sectors are highly essential. Many researches have been done on reducing the production time and cost, material wastage, mass production and quality range for components production. Additive manufacturing overcome all the problems and increase the production rate in a conceptual manner. AM is a layer-by-layer technology that can produce geometrical complex components in a single step process produced a drastic change in manufacturing.

Nearly all AM-components need post processing such as heat treatment in order to achieve the right properties such as surface quality, geometrical accuracy and mechanical properties. Surface heat treatment can include machining, blasting and polishing. Most of the materials always need a heat treatment, in the post 3D printing phase. To relieve stress on components, heat treatment is needed before their removal from the build plate. Among all the post processing method, the heat treatment process is of greater importance and significance. At high heat treatment temperatures, the heat treatment may minimize surface contamination, which may lead to a subsequent improvement in the mechanical response of the component. The materials mentioned above are mostly used for aerospace and medical applications in addition to these heat treatment benefits, in which to reduce surface contamination and to maximize mechanical properties, the rules have to be strictly followed.

Nitriding is a thermochemical surface treatment process where active nitrogen from the atmosphere enters into the surface of stainless steels at a defined temperature. During the nitriding process, there creates a compound layer and diffusion layer. The compound layer which is also called as white layer that mainly

consists of ε-$Fe_{2-3}N$ and traces of γ'-Fe_4N can provide better wear and corrosion resistance.

In a wide range of industrial heat-treating processes, including neutral hardening, liquid carburizing, liquid nitriding, austempering and martempering, salt baths are employed. Salt bath equipment is ideally suited for the heat treatment of ferrous and nonferrous alloys. The molten salt bath provides ready source of heat by conduction and distributed throughout the surface of the alloys. Salt baths are particularly effective heat treatment methods in which 93-97% of electrical energy is consumed by the salt bath that is directly supplied to heat the parts. Typically, ceramic-lined furnaces with submerged or immersed electrodes containing salts with a chloride structure are used to process steels that are heat treated in molten salts.

2. LITERATURE REVIEW

A literature survey was carried out based on the mechanical and microstructural properties and corrosion resistance of additively manufactured 17-4 PH steel alloy. Few studies on nitrided 17-4 PH steel on its mechanical and corrosion properties are also studied.

PH and maraging steels usually undergo age hardening treatment through precipitation are extensively suitable for fabrication through additive manufacturing. Both steels exhibit same strengthening behaviour. McGuire [1] proposed that reduction in carbon content for both the steels leads to the detrimental effect to corrosion resistance. On understanding the microstructure of PH steels by Ghaffari et al. [2], austenite phase appeared before quenching and after quenching martensitic phase remains at the room temperature with fewer amount of retained austenite phase. Stainless steel 17-4 PH is one of the most popular alloys in this category due to its high tensile and impact strength, high fracture toughness and high corrosion resistance at typical service temperatures below 300 ºC. 17-4 PH steels are the most commonly used PH steels due to its strengthening effect, printability and moderate corrosion resistance. The strengthening of low carbon martensitic stainless steels is achieved by the phase elements such as chromium, copper, nickel, nitrogen and molybdenum. For example, 15-5 PH steel is strengthened by copper, 13-8 PH steel by Nickel and aluminum and 17-4 PH is strengthened by copper precipitates.

Microstructural characterization includes grain size and grain morphology of components greatly depend on the temperature gradients, cool rates and thermal reheating cycles that are experienced during processing. LeBrun et al. [3] studied about the presence of austenite within the microstructure of 17–4 PH, and observed that the microstructure greatly affect the performance of 17–4 PH due to the transformation of austenite to martensite. Bayode et al. [4] studied about the scanning strategy on AM process influence the microstructure. Zhou et al. [5]and Jia and Gu. [6] studied about the laser energy density by changing the laser power parameters. Irrinki et al. [7] had a detailed study on the atomisation of 17-4 PH powder to analyse the densification and mechanical characteristics. The gas atomized and water atomized powders are used for the study which evident the gas atomized 17-4 PH steel showed a superior property on its microstructure, densification and mechanical strength.

Mechanical properties of 17-4PH stainless steel are better without post-processing when compared with the traditional method. Hu et al. [8] discussed about the solution treated condition, little evidence of martensite is present, and the aged condition shows no transformation to tempered martensite. Hardness vastly decreases during solutionizing and only increases marginally during aging. Additively manufacture 17-4PH stainless steel fabricated through SLM with both atomized in either argon or nitrogen atmosphere has been comparatively studied. The results show that the correspondingly fabricated through SLM sample show that SLM fabricated martensitic products exhibit hardness Rockwell C (HRC) of 30, while on aged at 482 °C gives an HRC of 43. As mentioned by Stoudt et al. [9], austenitic products do not exhibit age hardening. The solutionized and aged steels show better yield and ultimate strengths when compared with additively manufactured steels. Nezhadfar et al. [10] discussed about the steels that are built under nitrogen atmosphere, exhibit a combination of retained austenite and martensite phases and possess a better fatigue resistance to crack growth. Considering the comparative study on fatigue strength, wrought alloy shows slightly higher fatigue strength than additively fabricated steel. As studied by Yadollahi et al. [11], the steel samples that are built-in vertical direction show a significantly lower elongation at breakage than horizontal counterparts. From the study of Gao et al. [12], it is cleared that by controlling the process parameters, the overall performance of the process during additive manufacturing is controlled.

Corrosion is a major factor that is to be considered for stainless steels. Vukkum and Gupta [13] deliberated the corrosion performance of LPBF parts, which shows slightly higher corrosion performance than wrought samples. There are many factors that have to be considered on corrosion behaviour of AM 17-4 PH steels, including the precipitates, inclusions, grain size, grain boundary, etc. All these factors are depending upon the process parameters and feedstock selection. Irrinki et al. [14] exposed the corrosion behaviour of laser powder bed fusion (LPBF) and wrought 17-4PH under electrochemical test and immersion exposure in 0.6 M NaCl emphasise that LPBF fabricated samples. A better corrosion resistance is obtained for AM fabricated samples than wrought alloys. The corrosion rate increased for the AM steel is due to the presence of pores in the samples. The usability of different powders as a feedstock and laser power can also affect the corrosion resistance of resulting components.

On post processing, the steels showed increase in yield strength, ultimate tensile strength, compressive strength, and hardness. Afkhami et al. [15] proposed the heat-treated samples are less ductile when compared with as-built. The grains are equally distributed, and the heat treatment improves the hardness, yield strength, tensile strength, and percentage of elongation. Grain refinement through shot peening improves the physical and mechanical properties of 17-4PH stainless steel. Heat treatment improves the mechanical properties of the stainless steels. García et al. [16] and Múnez et al. [17]reported the H900 aging heat treatment process in the case of ferritic and austenitic steels improve the hardness. This heat treatment showed a phase transformation from Body-centered Cubic (BCC) to Face-centered Cubic (FCC) structure. Almathami and Brochu [18] proposed the phase change intrudes a property variation in the component. Also, shot peening improves the fatigue life of stainless steel, residual strength and produced refined crystal structure.

Nitriding is basically classified into gas, liquid and plasma nitriding. Tesi et al. [19] studied about the plasma nitriding which is an alternate solution of age hardening treatment. On comparing other conventional nitriding process (gas and plasma), the salt-bath nitriding treatment is considered as a low-cost method with many advantages including low treatment temperature, less duration, high degree of shape and dimensional stabilities, and reproducibility. The salt- bath solution used

potassium nitrate and/or sodium nitrate as a reactive agent for the nitriding process. The salt solution on heating undergoes chemical reaction and releases active nitrogen.

Wang et al. [20] studied about the effect of salt-bath nitriding temperature that influence the microstructure and mechanical properties of wrought 17-4 PH steel. Wang et al. [21] also reported the erosion-corrosion behaviour of 17-4 PH steel on varying the treating time from 4 to 40 hours. As per the report, the maximum values measured from the treated surface are observed to be approximate 1100 HV0.1 at 610 °C, which is about 3.7 times as hard as the untreated material (309 HV0.1). The decrease in surface hardness at higher temperature (650 °C) was accompanied by the formation of a significant amount of CrN phase. On nitriding above 610 °C, expanded martensite transformed into expanded austenite. But in the sample nitrided at 650 °C, the expanded austenite decomposed into α N and CrN. Shih et al. [22] reported the oxides and nitrides that form on nitriding of 304, 430 and 17-4 PH stainless steels. For a given nitriding condition (923 K for 2 h), the 430 stainless steel obtained the thinnest nitride film while the thickest was on the 17-4 PH stainless steel. The intensity of the Fe_2O_3 oxide, Fe phase and Cr–N bond phases was the highest in the nitride film on the 17-4 PH stainless steel among three samples. Moreover, the research works on studying the nitriding effect on wrought 17-4 PH steel is countable. Therefore, we have done a study on understanding the effect of nitriding on AM manufactured 17-4 PH steel at different nitriding temperature conditions.

3. RESEARCH GAP

- A very few studies are reported on studying the characterization of nitrided 17-4 PH steel especially need attention on nitrided AM 17-4PH steel.
- Studying the effect of nitriding on Mechanical properties of AM 17-4 PH steel is not attended.
- There is a need of attention on studying the nitriding effect on microstructural characterization of AM 17-4 PH steel.
- Effective research is needed on understanding the corrosion behavior of additively manufactured 17-4 PH steel with the nitrided AM 17-4 PH steel.

4. OBJECTIVES

The main objective of the work is to study the effect of nitriding on AM 17-4 PH steel at different temperatures.

- To study the microstructural characterization using SEM, EDS and XRD analysis on nitrided AM 17-4 PH steel.
- To study the mechanical properties such as Hardness and surface roughness of nitrided AM 17-4 PH steel.
- To study the corrosion properties of nitrided AM 17-4 PH steel through Electro chemical test under acidic and alkaline environment.
- To study the corrosion behaviour of nitrided AM 17-4 PH steel through Neutral salt spray test.

4.1 Proposed Methodology

The outline of the proposed methodology is as shown in Figure 1.

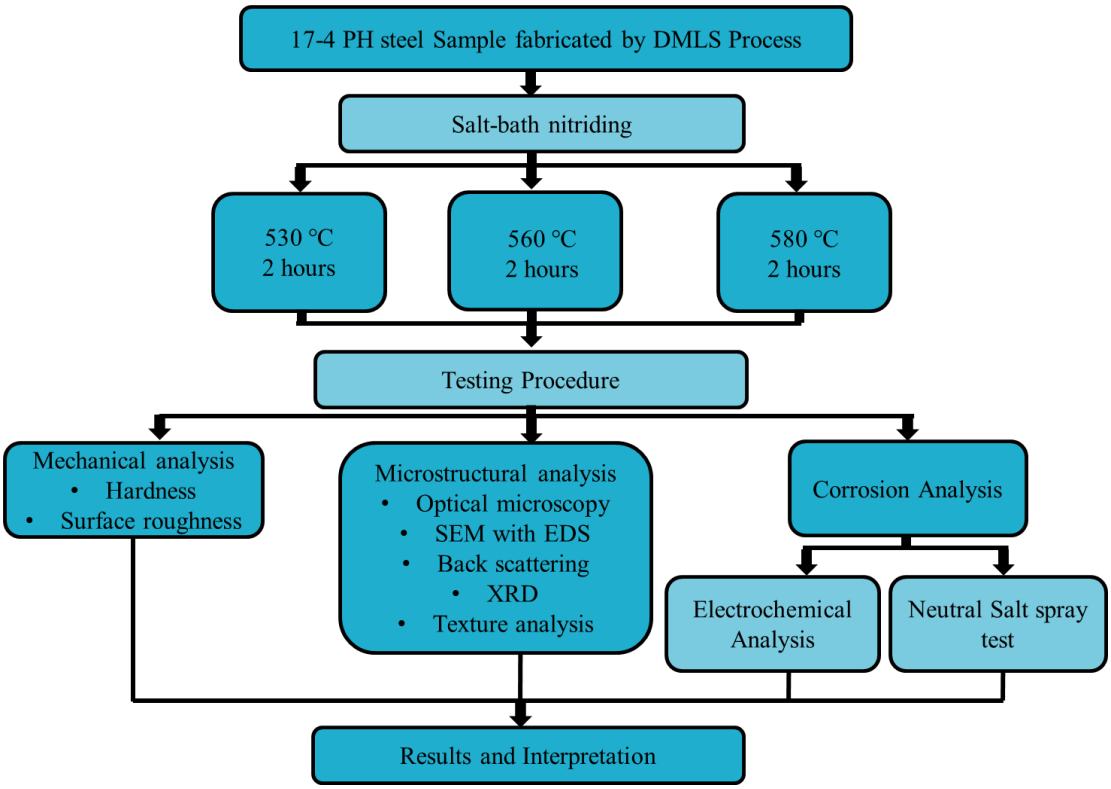


Figure 1: Research methodology.

4.2 Experimental Methodology

4.2.1 Material fabrication

17-4 PH steel, which is fabricated using the DMLS additive manufacturing process (EOS M 280) in an argon inert gas atmosphere, is selected for this study. The initial raw material used for the AM process is in powder form with a particle size ranging from 20 to 65 µm. Cylindrical samples of 100 mm in length and 15 mm in diameter are fabricated using the DMLS method with the following process parameters: platform temperature: 80°C; laser power source: 290 W; laser beam diameter: 80 µm and hatch distance: 0.1 mm.

4.2.2 Nitriding

Cylindrical samples of 5mm thick and 15mm diameter were cut from the AM rod. The axis of the cylindrical sample is parallel to the AM process build direction. The top flat surfaces of the cylindrical samples were ground and polished to a mirror finish and then ultrasonically cleaned. ASM handbook of Nitriding and ferritic carburizing recommends that the optimum temperature range for salt-bath nitriding of steels is 540°C to 580°C. Accordingly, in this work, salt-bath nitriding was performed at 530°C, 560°C, and 580°C for 2 hours. To remove the moisture and dirt, samples were preheated to 300°C for 30 minutes before the nitriding process. The preheated samples were then placed inside the cyanide-free salt-bath nitriding chamber at the predetermined temperatures of 530°C, 560°C, and 580°C for 2 hours. The samples were instantly water quenched after nitriding.

4.2.3 Mechanical Testing

The micro-hardness of both as-built and nitrided 17-4 PH steels was measured using a Shimadzu HMV G-series Vickers Microhardness tester. Following the ASTM standard E384, microhardness tests were performed using a diamond-shaped indenter under a load of 500 g and a dwell period of 10 seconds on the top surface and in the sample's cross-section. On the top surface, micro-hardness tests were performed at several locations for all samples. On the cross-sectional view, hardness tests were conducted with a spacing of 10 µm from the surface into the substrate. The surface roughness was measured using Surftest SJ-301 (Mitutoyo) profilometer at a scan speed of 0.25 mm/s at five different locations for each sample and repeated three times.

4.2.4 Microstructural studies

Grain morphology and the spatial distribution of precipitates are characterized using optical microscopy (OM) and SEM images. For microscopy, specimens were polished using the standard metallographic techniques and etched with Viella's reagent. The etching solution consists of 1gm Picric acid + 5 ml HCl + 100 ml ethanol. FESEM–CARLZEISS and EDAX made by BRUKER (USA) were used to image the topography of the top surface and cross-sectional microstructure of 17-4 PH steel. The X-ray diffraction technique is employed to identify the phases and measure the crystallographic texture. The phases were detected using a Bruker Eco D8 Advance X-ray diffractometer, which exerts Cu Kα radiation at an operating voltage of 40 kV and a current of 25 mA. X-ray diffraction patterns were obtained over a 2θ range of 30° to 100° with a step size of 0.02° with a counting time of 1s per step.

4.2.5 Corrosion Studies through Electro-chemical Test

The corrosion behaviours of as-built and nitrided 17-4 PH steel were evaluated by Potentiodynamic polarisation test in accordance with the ASTM G3-89 standard. The experiment was carried out in two different environments. The electrolyte solutions used were3.5 wt.% of sodium chloride (NaCl) and 0.5 M of sodium sulphate (Na2SO4). The electrochemical test was conducted in a three-electrode cell system. The reference electrode employed was silver/silver chloride (Ag/AgCl), while platinum foil was employed as a counter electrode. All tests were conducted at a room temperature with the constant scan rate of 1.0 mV/s. For anodic curves, a potential corresponding to current density value of 100 μA/cm² when its surface exposed to the electrolyte was 1.0 cm².To achieve a steady state condition, the open circuit potential (OCP) of the working electrode was measured before 60 minutes of potentio dynamic polarisation test. Tafel polarisation tests were carried out on the samples after 60 minutes of immersion on the electrolytic solution. All tests were conducted at a room temperature with the constant scan rate of 1.0 mV/s with the exposed area of 1.0 cm² to the electrolyte.

4.2.6 Corrosion Studies through Neutral salt spray Test

Neutral Salt Spray (NSS) is one of the universally accepted accelerated testing method conducted in controlled corrosive environment and provide authenticated data to correlate with long term exposure of component. In accordance with ASTM B117

standard, salt spray set-up contains 5 wt.% of NaCl solution and the test was performed by maintaining the chamber temperature at 35 ± 2 °C for 104 hours. The weight of the individual sample was recorded every 8 hours interval and the average weight loss was measured.

5. RESULTS AND DISCUSSION

5.1 Microstructural Analysis

To understand the observed improvement in the microhardness and surface roughness, in this section, microstructures of the top surface and cross-sections of the nitrided samples are analyzed compared to the as-built material. The top surface backscattered microstructure of the as-built and nitrided samples is shown in Figure 2. The dark spots in the microstructures are the precipitates, and it is observed in all the cases (Figure 2a-2d). The appearance of white lines (uneven chain-like pattern) indicates the formation of iron nitrides (Fe3-4N) on the surface during nitriding. The increase of dark regions in the 580°C case is most likely associated with the micro-segregation of grains due to the formation of precipitates.

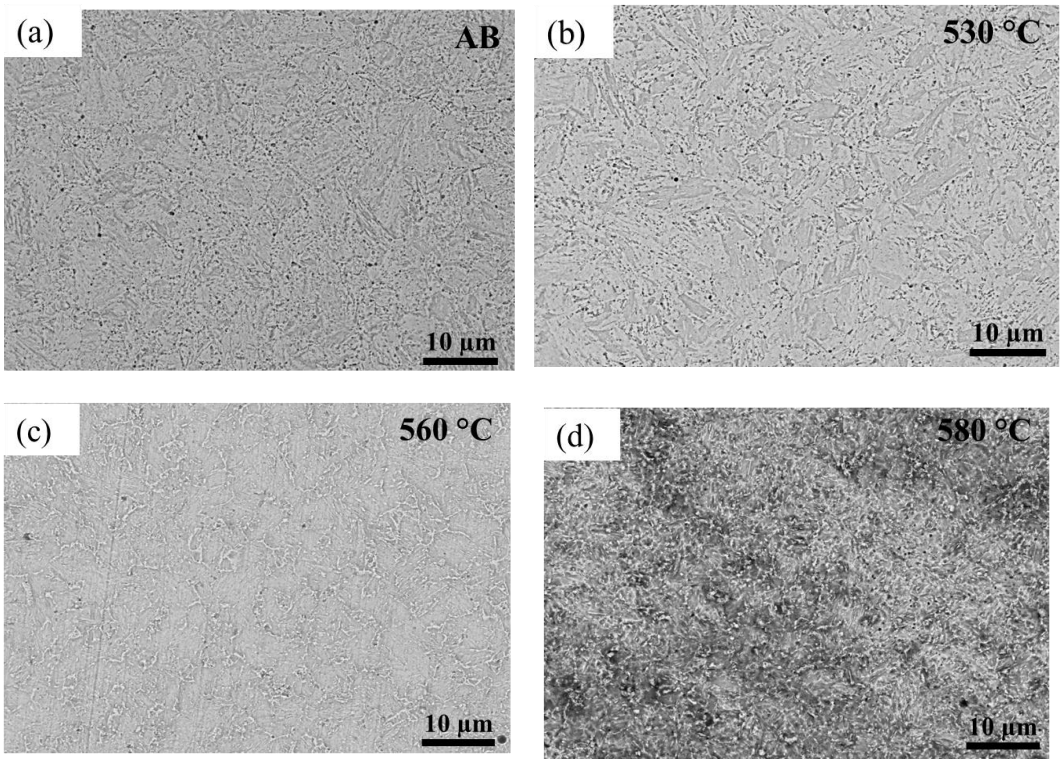


Figure 2: Backscattered microscopy images of the top surface of (a) As-built (AB) and nitrided at (b) 530 °C, (c) 560 °C, and (d) 580 °C 17-4 PH steel samples.

5.2 Mechanical Analysis

The microhardness values on the top surfaces and the cross-sections of the as-built and nitrided samples were measured using the diamond-shaped indenter. First, the as-built DMLS 17-4 PH steel exhibits better microhardness than the sintered/wrought material. The nitriding process improves the surface hardness significantly compared to the as-built material. Further, an increase in the nitriding temperature increases the microhardness values, but its effect is marginal.

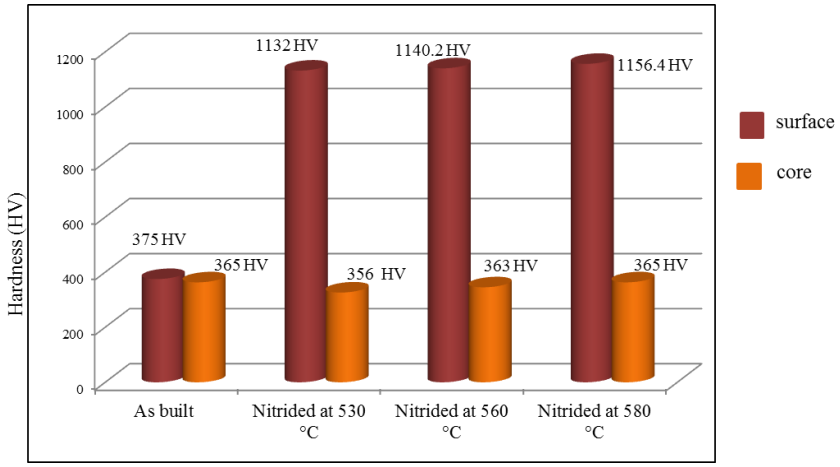


Figure 3: Comparative Hardness values for surface and core of as-built and nitrided 17-4 PH steel at different temperatures.

Upon nitriding, the microhardness value is higher at the surface for all three nitriding temperature cases and decreases rapidly first and then gradually while moving into the substrate. Finally, it reaches a value closer to the as-built case. The observed steep fall in the microhardness occurred across the interface between the nitride layer and transition layer/substrate, which can be easily seen from the marked nitride layer thickness lines. While the microhardness value for the as-built material and the nitrided 17-4 PH steel are almost constant from the surface to the substrate as shown in Figure 3. Among all three temperatures, microhardness values are higher for the 580°C case, and the rate of decrease is lower compared to the other two temperature cases. This suggests that at higher temperatures, i.e., 580°C, nitrogen diffusion happened beyond the identified nitride layer also.

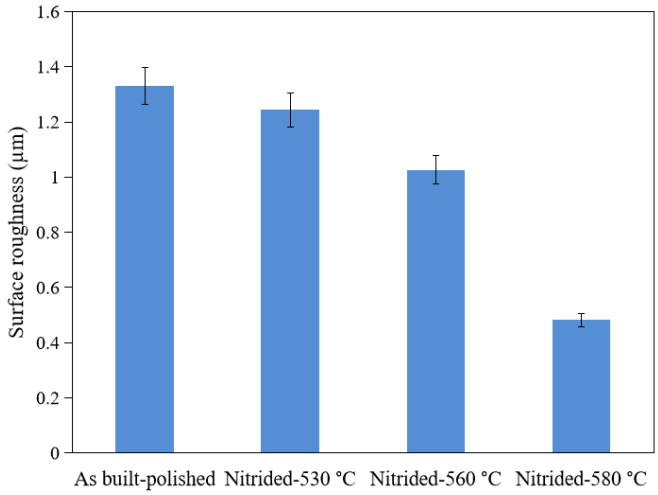


Figure 4. Effect of nitriding temperature on the surface roughness values of DMLS 17-4 PH steel.

Figure 4 shows the surface roughness of the as-built and nitrided samples measured using the surface profilometer. The measurement was repeated five times, and the observed variation was represented via error bars in Figure 4. The nitriding process decreases the surface roughness compared to the as-built sample. Further, the increase in nitriding temperature decreases the surface roughness values. Specifically, the surface roughness decreased from 1.330 µm of the as-built polished sample to 1.244 µm, 1.038 µm, and 0.482 µm corresponding to the samples nitrided at 530 °C, 560 °C, and 580 °C, respectively. The improvement in the surface roughness can be associated with the nitrogen diffusion process and also the possibility of recrystallization. Apart from the observed surface improvement, the variation in the surface roughness within a sample (error bar) decreases with increasing the nitriding temperature. It suggests that, in addition to the improvement in the average surface roughness, the nitriding process develops a homogeneous surface also.

5.3 Corrosion Behaviour

5.3.1 Electrochemical test

The electrochemical responses of 17-4 PH steel on the as-built and nitrided surfaces were studied by potentiodynamic polarisation tests under two different environments and the results obtained are subsequently discussed. In deaerated alkaline solutions, chloride accelerated the anodic kinetics of iron dissolution through a catalytic process (i.e) the metal atoms from the component dissolute as ions results

in pit formation. Pits growth is stopped by reducing the cathodic reaction to consume the electrons liberated by that is to polarize anodically. Considering both Figures (5a) and (5b), it is observed from as-built 17-4 PH steel specimen that there was no evidence of passive film. On increasing the corrosion current for 3.5 wt.% of NaCl solution, the absence of passive film led to the corrosion of metal. A highest current density of 6.68 mA/cm2 is obtained for as-built steel. The active chloride ions that are strongly negative charged in nature attack the passive film in defective zones, leading to the formation of localised pitting corrosion. Pitting is an autocatalytic process that has enough chlorides to keep the surrounding surface be passivized and allows the pit to grow.

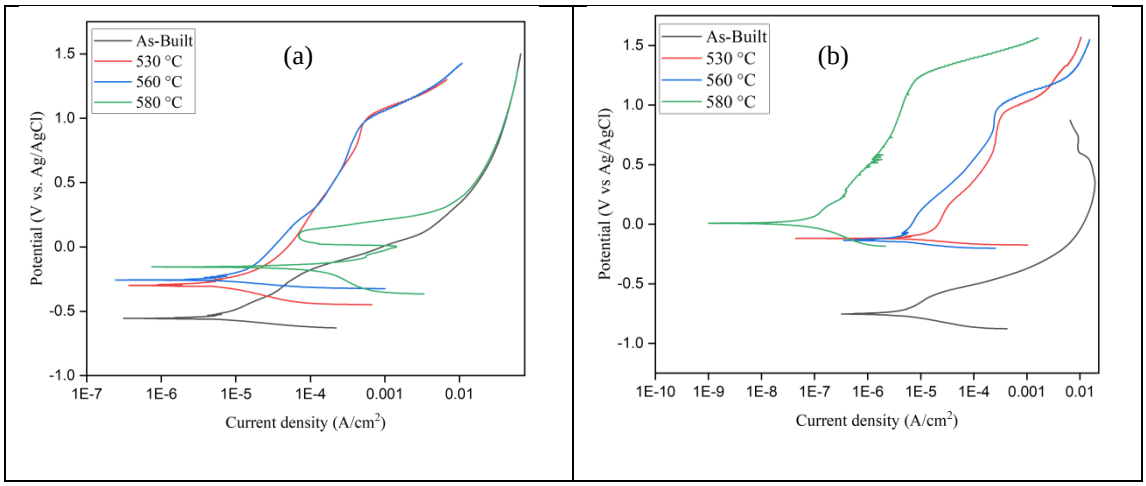


Figure 5: Potentiodynamic polarisation curves for 17-4 PH steel nitrided at different temperatures in (a) 3.5 wt.% of NaCl and (b) 0.5 M of Na2SO4 solutions.

Furthermore, the nitrided 17-4 PH steels at 530 °C, 560 °C and 580 °C showed better corrosion resistances than the as-built steel. Observing the results of nitrided steel at 530 and 560 °C, corrosion potentials (Ecorr) of -298 and -257 mV were obtained in NaCl environment behaviour with a sudden rise in current, respectively. It also showed a passive region to certain extent during the electrochemical test. The corrosion resistance was achieved by the formation of oxide layer of chromium rich oxide and iron rich hydroxides. The passive region exhibited complete breakdown of the passive film, results in uniform corrosion.

For AM 17-4 PH steel, nitrided at 580 °C showed passivation behaviour with a sudden rise in current density and also exhibited re-passivation behaviour.

Therefore, the highest corrosion potential of -155 mV with low corrosion current density (I_{corr}) of 1.14E-8 mA/cm2 was achieved. Similarly, the potentiodynamic polarisation curve of 0.5M of Na2SO4 solution is previously shown in Figure 5.4. The as-built 17-4 PH steel showed the lowest corrosion potential. A similar observation to that of NaCl condition was obtained, where there is no passivation film formed on as-built 17-4 PH steel in Na2SO4 environment. As the relationship between corrosion current and corrosion rate are correlated, the as-built steel showed the corrosion potential of -753 mV. The nitrided samples showed better improvement than as-built and wrought steel. The nitrided17-4 PH steels at 530 and 560 °C showed the potentials of -134 and -119 mV, respectively. Also, surface conditions of materials determine their corrosion resistances. The surface roughness of nitrided 17-4PH steel was comparatively less than that of as-built specimen. It was therefore evident from Figure.4 that at 580 °C, corrosion potential of 8.11 mV and a low Icorr value of 1.14E-8 mA/cm2 were obtained. As the pitting potential was towards positive, it showed more resistance to breakdown of passivation film. The presence of nitrogen in the nitrided steel led to re-passivation, which supressed the formation of pit.

5.3.2 Neutral salt spray test

The effects of varying nitriding temperatures of 530, 560 and 580 °C for two hours on corrosion resistance of additively manufactured (AM) 17-4 PH steels, using neutral salt spray (NSS) method for 104 hours. The Figure 6 shows the surface morphology of as-built and nitrided 17-4 PH steel before and after the salt spray test. The results of the analysis showed that a minimum weight loss was observed at 580 °C, due to the formation of passive oxide layer and nitrogen rich precipitates on the surface.

During oxidation, atmospheric oxygen reacted with surface components to form metal oxides. The metal oxides formed can dissolute after a period of time. Therefore, there is a possibility of weight gain or loss, due to the increased amount of metal oxides or dissolution of oxides. As-built sample corroded severely, due to fast oxide layer development at 24 hours. Rust formed after 56 hours for nitrided samples at 530 and 560 °C, while only minor rust formed after 96 hours at 580 °C. The weights loss after 104 hours were observed as 5.1×10-6, 1.7×10-6, 1.5×10-6 and 7×10-7 grams for as-built and nitrided samples at 530, 560 and 580 °C, respectively.

Continuous spraying of salt solution over sample surface caused oxide layer disintegration and deterioration. Active chloride ions from alkaline salt spray attacked weak zones, causing localised pitting corrosion. On the other hand, the nitrided steel exhibited minimum weight loss at 580 °C.

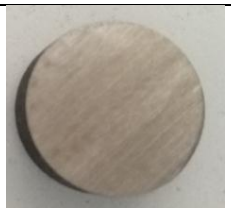
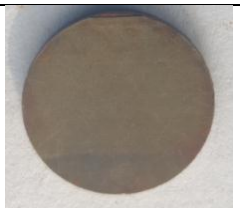
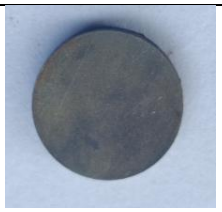
Time (hours)	17-4 PH steel			
	As-Built	Nitriding temperature (^{o}C)		
		530	560	580
Before corrosion				
After 104 hours				

Figure 6: Surface morphologies of as-built and nitrided 17-4 PH steel samples under salt spray corrosion for 104 hours.

6. RESEARCH FINDINGS

- The nitriding process increases the surface hardness approximately three times compared to the as-built condition.
- The surface roughness is reduced significantly by the nitriding process, and most importantly, it decreases more with an increase in the nitriding temperature.
- Detailed microstructural characterization reveals that the nitriding process (i) changes the columnar grain structure to more of an equiaxed; (ii) develops different types of inter and intra-granular precipitates; (iii) does not alter the initial weak crystallographic texture.

- The nitride layer formed on the surface of the stainless steel enhanced the surface properties. The nitride layer thickness of the samples obtained to be 12.01, 24.55 and 25.79 µm for 530, 560 and 580 °C, respectively.

- In NaCl and Na2SO4 solutions, 17-4 PH steel nitrided at 580 °C exhibited re-passivation behaviour with the highest corrosion potential of -155 mV and -753 mV.

- The as-built specimens showed corrosion in form of pits in both environments. Based on EDS analysis, the pit region showed the presence of Mn, S, Cu and O. Significantly, the nitrided 17-4 PH steel specimen the maximum corrosion resistance at 580 °C when compared with as-built and other nitrided steels in both environmental conditions. The improved corrosion resistance was due to the presence of homogeneous distribution of Cr and N on the surface along with other compounds.

- In Neutral salt spray test, the as-built steel showed more rust formation on its surface than the nitrided sample, whereas nitrided sample exhibited a better corrosion resistance at 580 °C.

- The corrosion rate of 4.423 mpy in as-built sample reduced to 0.606 mpy in nitrided sample at 580 °C. Chromium and iron nitrides acted as a barrier by forming a passive protective film that prevented the surface from corrosion.

7. ORGANIZATION OF THESIS

- Chapter 1: This chapter illustrates the detail study of precipitation hardened stainless steels, additive manufacturing process along the comparative approach with conventional techniques and surface treatments followed for stainless steel.

- Chapter 2: This chapter describes a detailed literature study on recent research on the additive manufacturing process of 17-4 PH steel is reported. Also, the effect of additive manufactured 17-4 PH components about its microstructure, mechanical, and corrosion properties were described. The research gap was initiated from the detailed literature survey and the objectives were framed. Based on the objectives the proposed research methodologies were developed.

- Chapter 3: This chapter explains the composition of 17-4 PH stainless steel and process parameters involved in the fabrication of the sample. Also, explains the procedure carried out on nitriding process. The evaluation of tests such as mechanical test- hardness and surface roughness test, microstructural characterization – Optical, SEM with EDS, XRD and crystallographic orientation were also discussed in detail.

- Chapter 4: This chapter described the effect of nitriding on mechanical and microstructural characterization of 17-4 PH steel and compared with the as-built steel. The detail study on microstructure with precipitation analysis is performed and discussed with the help of SEM images.

- Chapter 5: In this chapter, the corrosion behaviours of additively manufactured nitrided 17-4 PH steels in different environments were studied. The influence of nitriding on corrosion resistance of 3.5 wt.% of sodium chloride (NaCl) and 0.5M of sodium sulphate (Na2SO4) environments were investigated. The results are analysed through SEM and EDS mapping.

- Chapter 6: This chapter deals with the effects of varying nitriding temperatures of 530, 560 and 580 °C for two hours on corrosion resistance of additively manufactured (AM) 17-4 PH steels, using neutral salt spray (NSS) method for 104 hours. The microstructural analysis was done through SEM with EDS mapping and XRD. The weight estimation was carried out before and during corrosion test for every 8 hours.

- Chapter 7: Based on the results and discussion, the conclusions are made for the research work, and the future scope of the work was discussed in this chapter.

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LIST OF PUBLICATIONS

1. J. R. Jessy Michla, N. Rajini, S. O. Ismail, T. Ram Prabhu, S. Siengchin, and M. P. Indira Devi and F. Mohammad, Corrosion behaviours of additively manufactured nitrided17-4 PH steels in different environments, *Transactions of the Indian Institute of Metals*, Ms.No. TIIM-D-22-01216R1 (accepted).
2. J. R. Jessy Michla, N. Rajini, S. O. Ismail, T. Ram Prabhu, F. Mohammad, S. Siengchin, and M. P. Indira Devi, Effects of nitriding on salt spray corrosion resistance of additively manufactured 17-4 PH steels, *Materials Letters* **330** (2023), 133258.
3. J. R. Jessy Michla, B. Ravikumar, T. Ram Prabhu, S. Siengchin, M. Arul Kumar and N. Rajini, Effect of nitriding on mechanical and microstructural properties of Direct Metal Laser Sintered 17-4PH stainless steel, *Journal of Materials Research and Technology*, **19** (2022), 2810-2821.
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